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# *Indian Standard*

## METHODS FOR DETERMINATION OF STRENGTH OF NAPHTHOLS ( AZOIC COUPLING COMPONENTS ) ( GRAVIMETRIC AND VOLUMETRIC METHODS )

*( First Revision )*

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BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# Indian Standard

## METHODS FOR DETERMINATION OF STRENGTH OF NAPHTHOLS ( AZOIC COUPLING COMPONENTS ) ( GRAVIMETRIC AND VOLUMETRIC METHODS )

### ( First Revision )

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# *Indian Standard*

## METHODS FOR DETERMINATION OF STRENGTH OF NAPHTHOLS ( AZOIC COUPLING COMPONENTS ) ( GRAVIMETRIC AND VOLUMETRIC METHODS )

### *( First Revision )*

#### 0. FOREWORD

**0.1** This Indian Standard ( First Revision ) was adopted by the Indian Standards Institution on 11 December 1981, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

**0.2** This standard was first published in 1967. In implementation of the standard it was observed by the industry that the gravimetric method given in the standard did not give consistent results and some naphthols were not getting dissolved completely. It is expected that the revised method given in this standard would be applicable to all naphthols and consistent results would be obtained.

**0.3** Standards of Weights and Measures Act, 1976 stipulates the use of International System of Units in the country; in order to familiarize the industry with this system, the recommended SI units for use in the textile industry are given in Appendix B.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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#### 1. SCOPE

**1.1** This standard prescribes two methods, namely, gravimetric and volumetric, for determination of strength of naphthols. Both the methods are applicable to the naphthols given in Appendix A.

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\*Rules for rounding off numerical values ( revised ).

**1.2** Volumetric method prescribed in the standard is not applicable to the mixtures of naphthols.

**1.2.1** In case of dispute, the gravimetric method shall be used for determination of strength of naphthols.

## 2. SAMPLING

**2.1** Lot — All the containers of the same naphthols and of the same concentration delivered to one buyer against one despatch note, shall constitute a lot.

**2.2** Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from the lot shall be as given below:

<i>Lot Size</i>	<i>Sample Size</i>
Up to 100	3
101 to 300	4
301 to 500	5
501 and above	7

**2.3** From each container, draw small quantities of the naphthol by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 50 g.

## 3. QUALITY OF REAGENTS

**3.1** Unless specified otherwise, pure chemicals shall be employed in tests, and distilled water ( *see* IS : 1070-1977\* ) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

## 4. GRAVIMETRIC METHOD

### 4.1 Apparatus

**4.1.1** Beaker — of 1 000 ml capacity.

**4.1.2** Sintered Glass Funnel — of appropriate size:

- with pore size 15 to 40 microns ( G-3 size ), and
- with pore size 5 to 15 microns ( G-4 size ).

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\*Specification for water for general laboratory use ( *second revision* ).



## 4.2 Reagents

**4.2.1 Methanol** — Pure ( see IS : 517-1967\* ).

**4.2.2 Sodium Hydroxide Solution** — 30 percent ( *m/m* ) and 1 percent ( *m/m* ).

**4.2.3 Hydrochloric Acid** — 1 : 1 ( *v/v* ).

## 4.3 Procedure

**4.3.1** Weigh accurately about 1 g of naphthol sample and transfer to 1 000 ml dry beaker. Paste it with 10 ml methanol and add 1 ml of 30 percent sodium hydroxide and stir to dissolve. Add 400 ml warm ( about 50°C ) 1 percent sodium hydroxide solution and stir till the solution becomes clear. Filter through sintered glass funnel G-3, wash the residual material first with 3 to 5 ml methanol and finally with two 25 ml portion of warm 1 percent sodium hydroxide solution. Transfer the filtrate and washing quantitatively into a 1 000 ml beaker. Add while stirring with a glass rod, hydrochloric acid drop by drop until the solution is acidic to Congo red paper. Digest the precipitated mass on water-bath for about 2 to 3 hours. Allow the precipitate of naphthol to settle down. Filter through a sintered glass funnel G-4. Wash the precipitate with hot water till the filtrate is free from chlorides.

**4.3.2** Dry the residue at 100 to 110°C to constant weight and weigh.

**4.3.3** Calculate the percentage of naphthol by the following formula:

$$p = \frac{b}{a} \times 100$$

where

*p* = percentage, by mass of the naphthol under test;

*b* = mass, in g, of the residue of the naphthol; and

*a* = mass, in g, of the naphthol taken for test.

**4.4** Repeat the test twice as given in 4.3.1 and 4.3.2 and calculate the percentage of naphthol by the formula given in 4.3.3.

**4.5** Calculate the average of the values obtained as in 4.3.3 and 4.4.

## 5. VOLUMETRIC METHOD

### 5.1 Apparatus

#### 5.1.1 Amber-Coloured Jacketed Burette

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\*Specification for methanol ( methyl alcohol ) ( *first revision* ).

**5.1.2 Amber-Coloured Graduated Flask** — of 500 ml capacity.

**5.1.3 Mechanical Stirrer**

**5.1.4 Beakers** — of 1 000 and 500 ml capacities.

**5.1.5 Water-Bath**

## **5.2 Reagents**

**5.2.1 Para Amino Acetanilide Diazonium Chloride Solution (Diazo Solution)** — 0.05 N, prepared as follows:

Weigh accurately 3.77 g of para amino acetanilide and transfer it to a 500 ml beaker. Add 55 ml of 1 N hydrochloric acid and 100 ml of water. Cool externally with ice to 5 to 10°C while stirring mechanically. Add 25.5 ml of 1 N sodium nitrite solution in about 5 minutes times. Continue stirring for 10 minutes and test for excess of nitrite with starch iodide paper (see Note). Transfer the solution to a 500 ml amber-coloured graduated flask and make up the volume with ice cold water. Keep the diazo solution in ice-bath.

NOTE — The starch iodide paper should give a positive faint test of excess of nitrous acid

**5.2.2 Diazotized Aniline Solution** — 0.05 N, prepared as follows:

Weigh accurately 2.325 g of redistilled aniline and diazotize it in the same manner as for para amino acetanilide (see 5.2.1).

NOTE — This solution should be used for estimating the strength of C. I. Azoic Coupling Component 18.

**5.2.3 Dianisidine Diazonium Chloride** — 0.02 N, prepared as follows:

Diazotize 5 g to the dianisidine base with 10 ml of hydrochloric acid and 3 g of solid sodium nitrite. Neutralize the solution with a fresh sodium acetate solution and dilute it to 1 000 ml.

**5.2.4 H-Acid (1-Naphthol-3,6, Disulphonic Acid, 8 Amino)** — 0.1 percent, prepared as follows:

Dissolve 0.1 g of H-acid in 100 ml of water containing 5 ml of 2 N sodium carbonate.

**5.2.5 Hydrochloric Acid** — 1 N and 0.1 N.

**5.2.6 Sodium Hydroxide Solution** — 1 N.

**5.2.7 Dilute Sodium Hydroxide Solution** — 2.5 ml of 40 percent sodium hydroxide in 100 ml of water.

**5.2.8 Dilute Acetic Acid** — 25 percent (m/v).

**5.2.9 Pyridine**

### 5.3 Procedure

**5.3.1** Take about 1 g of naphthol from the composite sample. Weigh it accurately and dissolve it in 40 ml of dilute sodium hydroxide solution (see 5.2.7). Heat the solution, if necessary, to dissolve the naphthol completely. Cool and add dilute acetic acid till the pH of the solution is neutral (pH 6 to 7). Dissolve the precipitated naphthol in 80 ml of pyridine with constant stirring. Add more pyridine, if necessary. Cool it to 25°C and keep under mechanical agitation.

**5.3.2** Titrate the solution obtained as in 5.3.1 against diazotized para amino acetanilide solution (diazo solution) (see 5.2.1) from an amber-coloured burette equipped with a water jacket through which water at about 5°C is circulated. Spot a drop of the reaction mixture on a filter paper. Spot a drop each of dianisidine diazonium chloride solution and H-acid solution near the spot of the reaction mixture. Take the end-point as the mean of the two readings, one which gives a negative test for naphthol with dianisidine diazonium chloride and the other which gives a positive test for para amino acetanilide diazonium chloride solution with H-acid. Note the volume of the para amino acetanilide diazonium chloride solution required.

**NOTE 1** — The para amino acetanilide diazonium chloride solution should be added initially in portions of 1 to 2 ml, testing the reaction mixture after each addition for excess of diazonium chloride or naphthol.

**NOTE 2** — If the bleed of reaction mixture when spotted on filter paper is not clear, a small amount of solid common salt should be kept on filter paper and the drops of reaction mixture should be dropped on it.

**NOTE 3** — If naphthol is in excess, a colouration is seen at the junction of the spots of dianisidine diazonium chloride and the reaction mixture. If diazo solution is in excess, a pink colour is seen at the junction of the spot of H-acid and the reaction mixture. The addition of para amino acetanilide diazonium chloride solution should be in instalments of 0.2 to 0.3 ml near the end point.

**NOTE 4** — In case of Azoic Coupling Component 18, diazotized aniline solution (see 5.3.2) should be used.

**NOTE 5** — C. I. Azoic Coupling Component 5 is dissolved in pyridine at 40°C. No alkali is added. The solution is cooled to 5 to 10°C and titrated against diazotized para amino acetanilide.

**5.3.3** Calculate the strength of naphthol by the following formula:

$$P = \frac{0.005 \times M \times A \times 100}{B}$$

NOTE — In case of Azoic Coupling 5, the coupling takes place at two positions. Therefore, the amount of diazo solution consumed is double and hence the strength should be calculated as:

$$P = \frac{0.005 \times M \times A}{2} \times \frac{100}{B}$$

where

$P$  = percentage, by mass, of naphthol;

$M$  = molecular mass of naphthol under test ( see Appendix A );

$A$  = volume, in millilitres, of para acetanilide diazonium chloride solution ( or diazotized aniline solution ) required for titration; and

$B$  = mass, in g, of the naphthol taken for the test ( see 4.3.1 ).

5.4 Repeat the procedure twice prescribed in 5.3.1 and 5.3.2 and calculate the strength of naphthol in each case.

5.5 Calculate the average of the values obtained as in 5.3.3 and 5.4.

## 6. REPORT

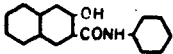
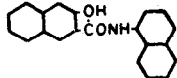
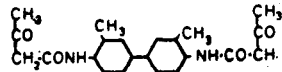
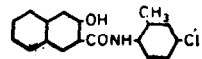
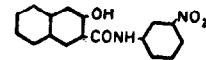
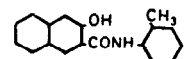
6.1 Report the value obtained as in 4.5 or 5.5 as the strength of naphthol under test.

6.2 Report also the method used, whether gravimetric or volumetric.

# APPENDIX A

( Clause 1.1 )

## GENERAL INFORMATION ABOUT ANALYSIS OF NAPHTHOLS

Sl. No.	*C. I. DESIGNATION OF NAPHTHOL	COMMON COMMERCIAL NAME OF NAPHTHOL	COLOUR INDEX No.	MOLECULAR MASS	STRUCTURAL FORMULA AND CHEMICAL NAME
(1)	(2)	(3)	(4)	(5)	(6)
i)	C. I. Azoic Coupling Component 2	Naphthol AS	C. I. 37505	263.0	
ii)	C. I. Azoic Coupling Component 4	Naphthol AS-BO	C. I. 37560	313.0	
iii)	C. I. Azoic Coupling Component 5	Naphthol AS-G	C. I. 37610	380.0	
iv)	C. I. Azoic Coupling Component 8	Naphthol AS-TR	C. I. 37525	311.5	
v)	C. I. Azoic Coupling Component 17	Naphthol AS-BS	C. I. 37515	308.0	
vi)	C. I. Azoic Coupling Component 18	Naphthol AS-D	C. I. 37520	277.0	

\*Colour Index ( 1956 ). Ed 2. Society of Dyers and Colourists, UK; and American Association of Textile Chemists and Colorists, USA.

## APPENDIX B

( Clause 0.3 )

## RECOMMENDED SI UNITS FOR TEXTILES

SL No.	CHARACTERISTIC	SI UNIT		APPLICATION
		Unit	Abbrevia- tion	
1.	Length	Millimetre	mm	Fibres
		Millimetre, centimetre	mm, cm	Samples, test speci- mens ( as appro- priate )
		Metre	m	Yarns, ropes, cordage, fabrics
2.	Width	Millimetre	mm	Narrow fabrics
		Centimetre	cm	Other fabrics
		Millimetre, centimetre	mm, cm	Samples, test specimens ( as appropriate )
				Carpets, druggets, <i>Durries</i> ( as appropriate )
3.	Thickness	Micrometre ( micron )	$\mu\text{m}$	Delicate fabrics
		Millimetre	mm	Other fabrics, carpets, felts
4.	Linear density	Tex	tex	Yarns
		Millitex	mtex	Fibres
		Decitex	dtex	Filaments, filament yarns
		Kilotex	ktex	Slivers, ropes, cordage
5.	Diameter	Micrometre ( micron )	$\mu\text{m}$	Fibres
		Millimetre	mm	Yarns, ropes, cordage

Sl No.	CHARACTERISTIC	SI UNIT		APPLICATION
		Unit	Abbrevia- tion	
6.	Circumference	Millimetre	mm	Ropes, cordage
7.	Threads in fabric:			Woven fabrics ( as appropriate )
	a) Lengthwise	Number per centimetre	ends/cm	
		Number per decimetre	ends/dm	
	b) Widthwise	Number per centimetre	picks/cm	
		Number per decimetre	picks/dm	
8.	Warp threads in loom	Number per centimetre	ends/cm	Reeds
9.	Stitches in knitted fabric:			Knitted fabrics ( as appropriate )
	a) Lengthwise	Courses per centimetre Courses per decimetre	courses/ cm courses/ dm	
	b) Widthwise	Wales per centimetre	wales/cm	
		Wales per decimetre	wales/dm	
10.	Stitch length	Millimetre	mm	Knitted fabrics, made-up items
11.	Mass per unit area	Grams per square metre	g/m <sup>2</sup>	Fabrics

SL No.	CHARACTERISTIC	SI UNIT		APPLICATION
		Unit	Abbreviation	
12.	Mass per unit length	Grams per metre	g/m	Fabrics
13.	Twist	Turns per centimetre Turns per metre	turns/cm turns/m	Yarns, ropes, cordages ( as appropriate )
14.	Test or gauge length	Millimetre, centimetre	mm, cm	Fibre, yarn and fabric specimens ( as appropriate )
15.	Breaking load	Millinewton	mN	Fibres, delicate yarns ( individual or skeins )
		Newton	N	Strong yarns ( individual or skeins ), ropes, cordage, fabrics
16.	Breaking length	Kilometre	km	Yarns
17.	Tenacity	Millinewton per tex	mN/tex	Fibres, yarns ( individual or skeins )
18.	Twist factor or twist multiplier	Turns per centimetre $\times$ square root of tex	turns/cm $\times \sqrt{\text{tex}}$	Yarns ( as appropriate )
		Turns per metre $\times$ square root of tex	turns/m $\times \sqrt{\text{tex}}$	
19.	Bursting strength	Newton per square centimetre	N/cm <sup>2</sup>	Fabrics



SL No.	CHARACTERISTIC	SI UNIT		APPLICATION
		Unit	Abbrevia- tion	
20.	Tear strength	Millinewton, newton	mN, N	Fabrics ( as appropriate )
21.	Pile height	Millimetre	mm	Carpets
22.	Pile density	Mass of pile yarn in grams per square metre per millimetre pile height	$\text{g/m}^2/\text{mm}$ pile height	Pile carpets
23.	Elastic modulus	Millinewton per tex per unit deformation	mN/tex/ unit defor- mation	Fibres, yarns, strands

# INDIAN STANDARDS

## ON

## DYESTUFFS

IS:

- 3859-1966 Method for determination of strength of water soluble azo dyes by reduction with titanium trichloride
- 4360-1967 Method for determination of strength of fast bases
- 4394-1967 Method for evaluating strength of homogeneous vat dyestuffs
- 4459-1967 Method for determination of strength of direct dyestuffs by dyeing test
- 4472 ( Part I )-1967 Methods for identification of the application classes of dyes on textile materials: Part I Cotton and other cellulosic fibres
- 4472 ( Part II )-1968 Methods for identification of application classes of dyes on textile materials: Part II Wool, silk and other protein fibres
- 4472 ( Part III )-1973 Methods for identification of application classes of dyes on textile materials: Part III Man-made fibres
- 4946-1968 Method for evaluation of strength and shade of naphthol
- 5970-1970 Method for estimation of strength ( vat content ) of solubilized vat dyestuffs
- 6526-1971 Method for evaluation of strength and shade of fast bases by dyeing test
- 7447-1974 Method for evaluating strength of reactive dyes ( dichlorotriazinyl type ) by dyeing test
- 7448-1974 Method for evaluating strength of reactive dyes ( monochlorotriazinyl type ) by dyeing test
- 7842-1975 Method for evaluating strength of reactive dyes ( vinyl sulphone type ) by dyeing test
- 7843-1975 Method for evaluating strength and shade of acid dyes by dyeing test
- 7844-1975 Method for evaluating strength and shade of chrome dyes by dyeing test
- 7845-1975 Method for evaluating strength of reactive dyes ( trichloropyrimidyl type ) by dyeing test

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